

## Data Validation Report

**Project/Site Name:** Omega Chemical OU1 Jan 2004 Split Sampling

**Sample Delivery Group (SDG):** 04021B

**Parameters:** Volatiles

**Method:** 8260B and 524.2

**Laboratory:** USEPA Region 9 Laboratory

**Samples:**

<u>Sample ID</u>	<u>Lab Sample ID</u>	<u>Collection Date</u>	<u>Matrix</u>
OC-OU1-1	0401063-01	01/20/04	Solid
OC-OU1-2	0401075-01	01/21/04	Solid
OC-OU1-3	0401075-02	01/21/04	Solid
OC-OU1-4	0401075-03	01/21/04	Water
OC-OU1-5	0401075-04	01/21/04	Solid

## **Introduction/Summary**

This data review report covers the sample delivery group and associated samples listed on the cover sheet. The analyses were per USEPA Method 8260B and 524.2. The quality assurance and quality control procedures (QA/QC) were per project specific sampling and analysis plan.

This review is based on the method and project approved QA/QC procedures and EPA data validation functional guidance; the following subsections correlate to these guidelines. The sections detail noted deviations if any. Tables summarizing all data qualification flags are provided at the end of this report. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from specified protocols (P) or is of a technical advisory nature due to sample matrix (A).

Data qualifiers, if any, are summarized at the end of this report.

## I. Holding Times

Samples were analyzed within 14 days (7 days if unpreserved) of collection as required.

All samples were analyzed within the holding times however it is noted that soil samples were received outside of the 48 hour sample preservation holding time. The soil sample OC-OU1-2 was collected on 01/21/04 at 07:45 and received on 01/23/04 at 10:47. The soil sample OC-OU1-3 was collected on 01/21/04 at 11:37 and received on 01/23/04 at 10:47. The soil sample OC-OU1-5 was collected on 01/21/04 at 17:22 and received on 01/23/04 at 10:47.

## II. GC/MS Instrument Performance Check

Instrument performance was checked prior to initial calibration and calibration verification. All ion abundance requirements were met for BFB as listed below:

<u>m/z</u>	<u>ION ABUNDANCE CRITERIA</u>
50	15.0 - 40.0% of m/z 95
75	30.0 - 80.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 - 120 % of m/z 95
175	5.0 - 9.0% of m/z 174
176	95.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

## III. Initial Calibration

An initial five-point calibration was performed using the required concentrations prior to sample analysis.

Percent relative standard deviations (%RSD) were less than 30% for CCCs and less than or equal to 15% for mean RSD for all analytes with no individual analyte %RSD greater than 30%.

Average relative response factors (RRF) for volatile system performance check compounds (SPCC) were equal to or greater than 0.30 (> 0.10 for bromoform, chloromethane, and 1,1-dichloroethane) with the exception of the following:

Calibration Date	Analyte	RRF	Affected Samples	Flag	A or P
01/21/04	Bromoform	0.098	OC-OU1-4	J	P

	1,1,2,2-Tetrachloroethane	0.141			
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Second-source calibration verification was not carried out after five-point initial calibration.

#### IV. Continuing Calibration

Continuing calibration was verified daily before sample analysis and every 12-hours of analysis time using mid-level standards.

The relative response factor (RRF) percent deviations were less than 20% for all CCCs and all calibration analytes were within  $\pm 20\%$  of the expected values.

The relative response factors (RRF) for system performance check compounds (SPCC) were greater than or equal to 0.30 ( $> 0.10$  for bromoform, chloromethane and 1,1-dichloroethane). The following had RRFs  $< 0.30$

Continuing Calibration Standard	Analyte	RRF	Affected Samples	Flag	A or P
01/23/04 (524.2)	bromoform 1,1,2,2-tetrachloroethane	0.098 0.131	OC-OU1-4	J	P

#### V. Blanks

Method blank analysis was performed at the frequency of once for every analytical batch.

The concentrations of analytes in the method blanks were less than the reporting limits, with no detections reported, with the following exceptions:

Blank (date)	Analyte	Concentration	Affected Samples	Flag	A or P
B4A0199- BLK1	Tetrachloroethene	4.5	none	Detects BJ	A

Tetrachloroethene was not reported for the samples so no samples are affected by this blank contamination.

There were no field blanks with this SDG.

## VI. System Monitoring Compounds

Surrogate compounds were added to all laboratory blanks, LCS, MS/MSD and field samples per project specifications.

All surrogate recoveries were within project specified control limits.

## VII. Matrix Spike/Matrix Spike Duplicates

The samples B4A0187-MS2 and B40187-MSD2 were the matrix spike (MS) and matrix spike duplicate (MSD) for the water sample within this SDG. All of the percent recoveries and relative percent differences were within control limits for precision and accuracy.

The samples B4A0161-MS2 and B4A0161-MSD2 were the matrix spike (MS) and matrix spike duplicate (MSD) for this SDG. All of the percent recoveries (%R) and relative percent differences (RPD) were within control limit for precision and accuracy with the following exceptions:

Analyte	%R MS	%R MSD	RPD	Affected Samples	Flag	A or P
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	58 %	38 %	42 %	OC-OU1-2 OC-OU1-5	J	A
Trichlorofluoromethane	80 %	62 %	25 %			
Dichloromethane	80 %	59 %	30 %			
trans-1,2-Dichloroethene	81 %	68 %	17 %			
1,1-Dichloroethane	75 %	38 %	65 %			
cis-1,2-Dichloroethene	78 %	15 %	135 %			
1,1,1-Trichloroethane	72 %	42 %	53 %			
1,1-Dichloropropene	84 %	68 %	21 %			
Toluene	76 %	61 %	22 %			
m&p-Xylene	78 %	69 %	12 %			
1,3-Dichlorobenzene	78 %	68 %	14 %			
1,4-Dichlorobenzene	78 %	68 %	14 %			
Tetrachloroethane	904 %	NR	NR	none	none	A

The tetrachloroethane should not have been reported in the MS as it was not in the MSD due to the high concentration of the analyte in the original result.

The samples B4A0190-MS2 and B4A0190-MSD2 were the matrix spike (MS) and matrix spike duplicate (MSD) for this SDG. All of the percent recoveries and relative percent differences were within control limit for precision and accuracy with the following exceptions:

Analyte	%R MS	%R MSD	RPD	Affected Samples	Flag	A or P
1,1-Dichloroethane	57 %	12 %	130 %	OC-OU1-3	J	A
1,2-Dichloroethane	42 %	19 %	75 %			
Chlorodibromomethane	142 %	138 %	3 %			
Dichloromethane	80 %	65 %	21 %			

### **VIII. Laboratory Control Sample (LCS)**

At least one laboratory control sample per analytical batch was analyzed.

All percent recoveries were within project specified control limits for precision and accuracy.

### **IX. Internal Standards**

Internal standards were added to all calibration standards, LCS, samples and blanks.

All internal standard retention times were within  $\pm 30$  seconds of the retention times of the latest daily calibration standard.

All internal standard area counts were within -50 percent to 100 percent of the midpoint of the initial calibration standard.

All retention times and internal standard area counts were within project specifications for precision and accuracy.

### **X. Compound Quantitation and Reporting Limits**

Compound quantitation algorithm was verified to be correct.

The MDLs have been provided by the laboratory on the sample reports along with the reporting limits. The laboratory has established method detection limits (MDLs) per 40 CFR Part 136 Appendix B. The laboratory MDLs are found to be consistent with project needs.

### **XI. Tentatively Identified Compounds (TICs)**

There were no tentatively identified compounds within this SDG.

### **XII. System Performance**

QC data at large indicate acceptable performance.

### **XIII. Overall Assessment of Data**

All data were found to be acceptable per specifications as noted above under introduction/summary with the exception of samples and analytes listed in the table at the end of this report, if any.

## Omega Chemicals OU1 Volatiles - Data Qualification Summary - SDG 04021B

SDG	Sample ID	Analyte	Flag	A or P*	Reason
04021B	OC-OU1-2 OC-OU1-3 OC-OU1-5	All	J	P	Sample Received outside of 48 hour sample preservation holding time.
04021A	OC-OU1-4	Bromoform 1,1,2,2-Tetrachloroethane	J	P	Initial Calibration RRF
04021A	OC-OU1-4	bromoform 1,1,2,2-tetrachloroethane	J	P	Continuing Calibration RRF
04021A	OC-OU1-2 OC-OU1-5	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113) Trichlorofluoromethane Dichloromethane trans-1,2-Dichloroethene 1,1-Dichloroethane cis-1,2-Dichloroethene 1,1,1-Trichloroethane 1,1-Dichloropropene Toluene m&p-Xylene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	J	A	Matrix spike/Matrix spike duplicate %R and /or RPD
04021A	OC-OU1-3	1,1-Dichloroethane 1,2-Dichloroethane Chlorodibromomethane Dichloromethane	J	A	Matrix spike/Matrix spike duplicate %R and /or RPD

\*P-Flag is due to deviation from criteria limits

A- Flag is expected to be due to sample matrix effects

## Omega Chemicals OU1 Volatiles - Blanks Data Qualification Summary - SDG 04021A

No data is qualified due to blank results.



## Data Validation Report

**Project/Site Name:** Omega Chemical OUI Jan 2004 Split Sampling

**Sample Delivery Group (SDG):** 040121B

**Parameters:** Semivolatiles

**Method:** EPA 8270C

**Laboratory:** EPA Region 9 Laboratory

**Samples:**

<u>Sample ID</u>	<u>Lab Sample ID</u>	<u>Collection Date</u>	<u>Matrix</u>
OC-OU1-1	0401063-01	01/20/04	Solid
OC-OU1-2	0401075-01	01/21/04	Solid
OC-OU1-3	0401075-02	01/21/04	Solid
OC-OU1-4	0401075-03	01/21/04	Water
OC-OU1-5	0401075-04	01/21/04	Solid

## **Introduction/Summary**

This data review report covers the sample delivery group and associated samples listed on the cover sheet. The analyses were per USEPA Method 8270C. The quality assurance and quality control procedures (QA/QC) were per project specific sampling and analysis plan.

This review is based on the method and project approved QA/QC procedures and EPA data validation functional guidance; the following subsections correlate to these guidelines. The sections detail noted deviations if any. Tables summarizing all data qualification flags are provided at the end of this report. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from specified protocols (P) or is of a technical advisory nature due to sample matrix (A).

Data qualifiers, if any, are summarized at the end of this report.

## I. Holding Times

Samples were extracted within 7 days (water) or 14 days (soil) of collection as required. Analyses were performed within 40 days after extraction. All samples were within project specifications.

## II. GC/MS Instrument Performance Check

Instrument performance was checked prior to initial calibration and calibration verification. All ion abundance requirements were met for DFTPP as listed below:

<u>m/z</u>	<u>ION ABUNDANCE CRITERIA</u>
51	30.0 - 60.0% of m/z 198
68	Less than 2% of m/z 69
69	0.0 – 100% of m/z 198
70	Less than 2% of m/z 69
127	40.0 - 60.0% of m/z 198
197	Less than 1% of m/z 198
198	Base peak, 100% relative abundance
199	5.0 - 9.0% of m/z 198
275	10.0 -30.0% of m/z 198
365	Greater than 1% of m/z 198
441	Present, but less than m/z 443
442	Greater than 40.0% of m/z 198
443	17.0 - 23.0% of m/z 442

## III. Initial Calibration

An initial five-point calibration was performed using the required concentrations prior to sample analysis.

Percent relative standard deviations (%RSD) were less than 30% for CCCs and less than or equal to 15% for mean RSD for all analytes with no individual analyte %RSD greater than 30%.

Calibration Date	Analyte	% RSD	Associated Samples	Flag	A or P
01/19/04	Di-n-octylphthalate	37.10 %	None	J	P

No further action was recommended since this SDG was only analyzed for 1,4 Dioxane.

Average relative response factors (RRF) for volatile system performance check compounds (SPCC) were equal to or greater than 0.05.

Second-source calibration verification (SSCV) was carried out once per five-point initial calibration. All analytes were within  $\pm 25\%$  of the expected values, with the following exception:

Calibration ID	Analyte	%D	Associated Samples	Flag	A or P
0401006-SCV1	Benzyl alcohol	41.3 %	None	J	P

No further action was recommended since this SDG was only analyzed for 1,4 Dioxane.

#### IV. Continuing Calibration

Continuing calibration was verified daily before sample analysis and every 12-hours of analysis time using mid-level standards.

The relative response factor (RRF) percent deviations were less than 20% for all CCCs and all calibration analytes were within  $\pm 20\%$ , with the following exception:

Calibration Date	Analyte	%D	Associated Samples	Flag	A or P
01/27/04	Di-n-octyl phthalate	25.9 %	None		
02/06/04	Di-n-octyl phthalate	30.3 %	None		
02/06/04	Hexachloropentadiene	79.4 %	None		
	Di-n-octyl phthalate	32.8 %			

No further action was recommended since this SDG was only analyzed for 1,4 Dioxane.

The relative response factors (RRF) for system performance check compounds (SPCC) were greater than or equal to 0.05.

The samples were analyzed on January 21 and 23 of 2004. There were no continuing calibration standards or sample analyzed on these dates in the raw data.

#### V. Blanks

Method blank analysis was performed at the frequency of once for every analytical batch.

The concentrations of analytes in the Method Blank were less than the reporting limits, with no detections.

#### VI. System Monitoring Compounds

Surrogate compounds were added to all laboratory blanks, LCS, MS/MSD and field samples per project specifications.

All surrogate recoveries were within project specified control limits for precision and accuracy with the following exceptions:

Surrogate	%R	Associated Samples	Flag	A or P
1,4-Dioxane-d8	35 %	OC-OU1-1	J	A
1,4-Dioxane-d8	34 %	OC-OU1-2	J	A
1,4-Dioxane-d8	35 %	OC-OU1-3	J	A
1,4-Dioxane-d8	35 %	OC-OU1-5	J	A

## **VII. Matrix Spike/Matrix Spike Duplicates**

Sample OC-OU1-2 was used for the matrix spike and matrix spike duplicate. The %recoveris (%R) and relative percent differences (RPD) were within the project specific control limits.

## **VIII. Laboratory Control Sample (LCS)**

At least one laboratory control sample per analytical batch was analyzed.

All % recoveries (%R) were within project specified control limits for precision and accuracy.

## **IX. Internal Standards**

Internal standards were added to all calibration standards, LCS, samples and blanks.

All internal standard retention times were within  $\pm 30$  seconds of the retention times of the latest daily calibration standard.

All internal standard area counts were within -50 percent to 100 percent of the midpoint of the calibration standard.

## **X. Compound Quantitation and Reporting Limits**

Compound quantitation algorithm was verified to be correct.

The MDLs have been provided by the laboratory on the sample reports along with the reporting limits. The laboratory has established method detection limits (MDLs) per 40 CFR Part 136 Appendix B. The laboratory MDLs are found to be consistent with project needs.

## **XI. Tentatively Identified Compounds (TICs)**

TICs reports were not required for this SDG.

## **XII. System Performance**

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The data at-large for target compounds indicate acceptable system performance

### **XIII. Overall Assessment of Data**

All data were found to be acceptable per specifications as noted above under introduction/summary with the exceptions of the samples and analytes listed in the table at the end of this report, if any.

**Omega Chemical OUI Semivolatiles - Data Qualification Summary - SDG #04021B**

<b>SDG</b>	<b>Sample</b>	<b>Analyte</b>	<b>Flag</b>	<b>A or P*</b>	<b>Reason</b>
04021B	OC-OU1-1 OC-OU1-2 OC-OU1-3 OC-OU1-5	1,4-Dioxane-d8	J	A	Surrogate Recoveries

\*P-Flag is due to deviation from criteria limits

A- Flag is expected to be due to sample matrix effects

**Omega Chemical OUI Semivolatiles - Blanks Data Qualification Summary – #04021B**

No blank detects were reported.